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Description

The present invention relates to a process for preparing maltose powder, specifically, to a process for preparing a stable maltose powder containing crystalline beta-maltose hydrate.

As disclosed, for example, in Japanese Patent Publication No. 3,937/79 and Japanese Patent Laid-Open No. 92,299/85, maltose powders containing crystalline beta-maltose hydrate have been manufactured by concentrating a high-purity maltose liquid to about 70-80 w/w % (moisture content of 20-30 w/w %), adding a seed crystal to the syrup, spray-drying a massecuite wherein crystallization of beta-maltose hydrate has proceeded to 30-50%, and ageing the resultant powder to a moisture content of 6 w/w %.

Conventional processes, however, have the drawback that they consume a relatively large amount of energy for drying at ambient temperature a maltose syrup having a relatively high moisture content (i.e. 20-30 w/w %) wherein crystallization of beta-maltose hydrate has been initiated by the addition of a seed crystal and this increases the manufacturing cost of maltose powder, and in addition to the disadvantage that a vigorous heating during the drying undesirably melts the resultant crystalline beta-maltose hydrate to hinder the attainment of a consistently high-quality maltose powder.

In order to overcome these drawbacks of the conventional processes, the present inventors studied various conditions for crystallizing beta-maltose hydrate in a syrup having the highest possible concentration. As a result, the present inventors found that the crystallization rate at ambient temperature is not necessarily increased as the saturation degree in the syrup is elevated; as well as that the crystallization rate is maximized when the moisture content of the syrup is in the range of 20-30 w/w % and a moisture content out of this range retards the crystallization rate.

Also was found that crystallization of beta-maltose hydrate in a high-concentration syrup having a moisture content below 10 w/w %, specifically, about 5-8 w/w %, which is comparable to that of commercial maltose powder is not recommendable in industrial-scale preparation of maltose powder.

While, as disclosed in Japanese Patent Laid-Open No.35,800/86, it has been known that a syrup having a moisture content below 10 w/w % tends to yield crystalline alpha-maltose.

By utilizing this, the present inventors discovered that the crystallization of beta-maltose hydrate can be accelerated by partially crystallizing anhydrous alpha-maltose in a high-concentration syrup having a moisture content below 10 w/w %, preferably, about 5-8 w/w %, to increase the moisture

content in its remaining amorphous part. Based on an additional finding that ageing of a crystalline alpha-maltose containing massecuite accelerates and facilitates both crystallization of beta-maltose hydrate and conversion of the crystalline alpha-maltose into crystalline beta-maltose hydrate, the present inventors established a novel process that enables industrial-scale preparation of a stable powder containing crystalline beta-maltose hydrate from a high-concentration syrup having a moisture content below 10 w/w %.

Accordingly the present invention provides a process for preparing maltose powder, comprising concentrating an aqueous solution of a high-purity maltose having a maltose content of at least 85 w/w %, on a dry substance basis, into a high concentration syrup having a moisture content below 10 w/w %;

allowing the resultant high-concentration syrup first to crystallize alpha-maltose in the presence of a seed crystal; and

allowing the resultant mixture to crystallize beta-maltose hydrate while converting the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate.

The wording "high-purity maltose" used in this specification means maltose having a maltose content of at least 80% DS (dry substance), preferably, 85% DS in order to obtain a satisfactorily stable maltose powder. To prepare such high-purity maltose from starch, a method as disclosed, for example, in Japanese Patent Publications Nos. 11,437/81 and 17,078/81, wherein gelatinized-or liquefied-starch is subjected to the action of beta-amylase and the released maltose is separated from polymer dextrans; and a method as disclosed, for example, in Japanese patent Publications Nos. 13,089/72 and 3,938/79, wherein gelatinized-or liquefied-starch is subjected to beta-amylase and a starch debranching enzyme such as isoamylase and beta-amylase are employable.

The maltose content of the obtained high-purity maltose is augmentable by subjecting the contaminant saccharides, such as maltotriose, to an enzyme as disclosed, for example, in Japanese Patent Publications Nos.28,153/81, 3,356/82 and 28,154/81, or by removing the contaminant saccharides with a fractionation as disclosed, for example, in Japanese Patent Laid-Open No.23,799/83 using a column of strongly-acidic cation exchange resin. Such fractionation can be effected by the fixed bed-, moving bed- or simulated moving bed-method.

To concentrate an aqueous solution of the obtained high-purity maltose having a maltose content of at least 80% DS, preferably, 85% DS or higher, to a high-concentration syrup, desirably, the lowest possible cost procedure, for example, concentra-

tion in vacuo, is employed.

Such aqueous solution is prepared into a high-concentration syrup having a moisture content below 10 w/w %, preferably, about 5-8 w/w %, which is first kept at a temperature in the range of 50-130 °C in the presence of a seed crystal to partially crystallize alpha-maltose, then aged at a temperature in the range of 10-70 °C to crystallize beta-maltose hydrate while converting the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate. The present inventors found that, when added to a syrup having a moisture content of 10 w/w % or higher, specifically, 12 w/w % or higher but lower than 25 w/w %, crystalline alpha-maltose dissolves in the syrup and substantially does not crystallize it, as well as that beta-maltose hydrate is much more crystallizable in such syrup.

Also was found that the presence of crystalline alpha-maltose in a high-concentration syrup having a moisture content below 5 w/w % is unfavorable because it requires addition of water to convert the crystalline alpha-maltose into crystalline beta-maltose hydrate.

An appropriate temperature for crystallizing alpha-maltose is 50-130 °C, preferably, 60-120 °C. An appropriate temperature for crystallizing beta-maltose hydrate and for converting crystalline alpha-maltose into crystalline beta-maltose hydrate is 10-80 °C, preferably, 20-70 °C.

Seed crystals may be added to accelerate the crystallization of maltose: Crystalline alpha-maltose, preferably, a mixture of crystalline alpha-maltose and crystalline beta-maltose hydrate is added as the seed crystal to a high-concentration syrup of a high-purity maltose in an amount of 0.001-20% DS, preferably, 0.1-5% DS, for example, by contacting, mixing and kneading.

To prepare the resultant syrup into a powder containing crystalline beta-maltose hydrate, for example, extrusion granulation and block pulverization are employable. In the case of the extrusion granulation, for example, while keeping at a temperature in the range of 60-120 °C, a high-concentration syrup of a high-purity maltose having a moisture content below 10 w/w % is kneaded together with a mixture of crystalline alpha-maltose and crystalline beta-maltose hydrate to effect a partial crystallization of alpha-maltose, and the resultant is fed to an extrusion granulator to obtain a granular masseccuite or a granular powder which is then aged at a temperature in the range of 20-70 °C to crystallize beta-maltose hydrate and also to convert the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate.

Alternatively, such a high-concentration syrup is kneaded together with a crystalline alpha-maltose seed while keeping at a temperature in the range of 60-120 °C, and the resultant mixture is

passed through an extrusion granulator while accelerating crystallization of alpha-maltose. The obtained granular masseccuite is allowed to contact with a crystalline beta-maltose hydrate seed, and then aged at a temperature in the range of 20-70 °C to accelerate both crystallization of beta-maltose hydrate and conversion of the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate. Thus, a maltose powder containing crystalline beta-maltose hydrate is obtainable.

In the block pulverization, for example, a high-concentration syrup of a high-purity maltose having a moisture content below 10 w/w % is placed in a crystallizer, and mixed with a blend of crystalline alpha-maltose and crystalline beta-maltose hydrate while accelerating crystallization of alpha-maltose by keeping at a temperature in the range of 60-120 °C. The resultant masseccuite is then transferred in a plastic tray, aged and solidified at a temperature in the range of 20-70 °C. The resultant block is cut and scraped with a cutting machine and/or a hammer mill to obtain a maltose powder containing crystalline beta-maltose hydrate. If necessary, moisture controlling, dehydrating and/or screening steps can be provided before or after the pulverizing step.

Since the obtained maltose powder having a moisture content approximately equal to that of the starting high-concentration syrup requires no or much less energy for postcrystallization drying, a consistently high-quality maltose powder can be manufactured at a reduced drying cost.

The mildly sweet white powder thus obtained is advantageously usable as a sweetener in various foods and beverages, as well as a humectant, vehicle or stabilizer in cosmetics, toiletries, pharmaceuticals and chemicals.

Several embodiments of the present invention will hereinafter be explained.

Example 1

A liquefied starch solution having a DE (Dextrose Equivalent) of about 0.5 was prepared by adding to a suspension of 1 part by weight of potato starch in 10 parts by weight of water a commercial bacterial liquefying alpha-amylase (EC 3.2.1.1), heating the mixture to 90 °C to effect gelatinization, and further heating it quickly to 130 °C to suspend enzymatic reaction. To the solution was added 100 units/g starch of isoamylase (EC 3.2.1.68) prepared from a culture of *Pseudomonas amyloclavata* ATCC 21262, and 50 units/g starch of "#1500", a beta-amylase (EC 3.2.1.2) derived from soybean, commercialized by Nagase & Company, Ltd., Osaka, Japan, and the resultant mixture was saccharified at pH 5.0 for 40 hours to obtain a high-purity maltose having a

maltose content of 92.5% DS. The high-purity maltose was then purified by carbon decolorization and resin refining, and concentrated in vacuo to obtain a high-concentration syrup having a moisture content of 6.5 w/w %. The syrup was then placed in a kneader, and added with 1% DS crystalline alpha-maltose and 1% DS crystalline beta-maltose hydrate while keeping at 95 °C. The resultant mixture was then kneaded for 3 minutes at this temperature, extruded in sheet shape, aged at 80 °C for 3 hours, further aged at 40 °C for 48 hours, and pulverized to obtain a maltose powder containing crystalline beta-maltose hydrate, moisture content of about 6.0 w/w %, in a yield of about 94% DS against the starting starch.

The product in the form of a non-hygroscopic stable powder is advantageously usable as a sweetener having a perceived sweetness value of about 1/3 compared to sucrose in a variety of foods and beverages.

Furthermore, the product is advantageously usable as a humectant, vehicle or stabilizer in cosmetics, toiletries, pharmaceuticals and chemicals.

Example 2

An aqueous solution of a high-purity maltose having a maltose content of 92.5% DS, obtained by the method in Example 1, was prepared into a high-concentration syrup having a moisture content of 5.8 w/w %. The syrup was then mixed with 2% DS crystalline alpha-maltose, and the mixture was granulated with an extrusion granulator. After ageing at 70 °C for 5 hours, the resultant granules were added with 2% DS crystalline beta-maltose, and the mixture was aged at 40 °C for 30 hours to obtain a maltose powder containing crystalline beta-maltose hydrate, moisture content of 5.3 w/w %, in a yield of about 95% DS against the starting starch.

Similarly as the product in Example 1, the product in the form of a stable powder free of moisture uptake is advantageously usable in foods, beverages, cosmetics, toiletries and pharmaceuticals.

Example 3

A suspension of 2 parts by weight of corn starch in 10 parts by weight of water was added with a commercial bacterial alpha-amylase, and the mixture was heated to 93 °C to effect liquefaction, followed by heating to 130 °C to suspend enzymatic reaction. The resultant liquefied starch solution having a DE of about 2 was quickly cooled to 55 °C, and then added with isoamylase (EC 3.2.1.68) and a soybean beta-amylase in respective amount of 120 units/g starch and 100 units/g

starch. The mixture was kept at pH 5.0 for 36 hours to effect saccharification, purified and concentrated similarly as in Example 1 to obtain a high-concentration syrup having a maltose content of about 88.2% DS and a moisture content of 6 w/w %. The syrup was then placed in a crystallizer, and added with 1% DS crystalline alpha-maltose seed and 1% DS crystalline beta-maltose hydrate seed at 90 °C. After mixing for 5 minutes while keeping at this temperature, the resultant was transferred to plastic trays, and aged first at 70 °C for 10 hours then at 40 °C for 48 hours to obtain a masseccuite solid in block shape. The masseccuite solid was then cut and scraped with a pulverizer, and screened to obtain a maltose powder containing crystalline beta-maltose hydrate, moisture content of about 5.5 w/w %, in a yield of about 92% DS against the starting corn starch.

The masseccuite solid was free of deformation and cracking, and exerted a satisfactory pulverizability.

Similarly as the product in Example 1, the product in the form of a stable powder free of moisture uptake is advantageously usable in foods, beverages, cosmetics, toiletries and pharmaceuticals.

As described above, the present invention relates to a process for preparing a maltose powder containing crystalline beta-maltose hydrate from a high-concentration syrup having a moisture content below 10 w/w % which has been deemed hardly crystallizable. More particularly, the preparation of such maltose powder is facilitated by concentrating an aqueous solution of a high-purity maltose having a maltose content above 85 w/w % into a high-concentration syrup having a moisture content below 10 w/w %, crystallizing alpha-maltose in the presence of a crystalline alpha-maltose seed, and crystallizing beta-maltose hydrate while converting the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate.

Since in the invention the postcrystallization drying can be carried out with no or much less amount of energy by concentrating in vacuo a high-concentration syrup to a moisture content approximately equal to a desired end product and this cuts a large amount energy for drying, consistently high-quality maltose powders are obtainable at a reduced drying cost. Thus, the present invention is very significant in the art.

The maltose powder obtained in this way is advantageously and extensively usable as a sweetener, humectant, vehicle or stabilizer in foods, beverages, cosmetics, toiletries, pharmaceuticals and chemicals.

Claims

1. A process for preparing maltose powder, comprising concentrating an aqueous solution of a high-purity maltose having a maltose content of at least 85 w/w %, on a dry substance basis, into a high-concentration syrup having a moisture content below 10 w/w %;
allowing the resultant high-concentration syrup first to crystallize alpha-maltose in the presence of a seed crystal; and
allowing the resultant mixture to crystallize beta-maltose hydrate while converting the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate.
2. A process according to claim 1, wherein said high-concentration syrup has a moisture content in the range of 5.0-8.0 w/w %.
3. A process according to claim 1 or 2, wherein crystalline alpha-maltose is used alone or in combination with crystalline beta-maltose hydrate as the seed crystal.
4. A process according to claim 1, 2 or 3, wherein crystallization of alpha-maltose is effected at a temperature in the range of 60-120 °C, while crystallization of beta-maltose hydrate and conversion of the resultant crystalline alpha-maltose into crystalline beta-maltose hydrate are effected at a temperature in the range of 20-70 °C.

Patentansprüche

1. Verfahren zur Herstellung von Maltosepulver, bei dem eine wäßrige Lösung einer hochreinen Maltose mit einem Maltosegehalt von mindestens 85 Gew.-% auf einer Trockensubstanzbasis in einen hochkonzentrierten Sirup mit einem Feuchtigkeitsgehalt unterhalb 10 Gew.-% konzentriert wird;
man zuerst ein Kristallisieren des erhaltenen hochkonzentrierten Sirups zu Alpha-Maltose in Anwesenheit eines Impfkristalls ermöglicht; und
man ein Kristallisieren der erhaltenen Mischung zu Beta-Maltosehydrat ermöglicht, während die erhaltene kristalline Alpha-Maltose in kristallines Beta-Maltosehydrat umgesetzt wird.
2. Verfahren nach Anspruch 1, bei dem der hochkonzentrierte Sirup einen Feuchtigkeitsgehalt im Bereich von 5,0 bis 8,0 Gew.-% aufweist.

3. Verfahren nach Anspruch 1 oder 2, bei dem kristalline Alpha-Maltose allein oder in Kombination mit kristallinem Beta-Maltosehydrat als Impfkristall verwendet wird.

4. Verfahren nach Anspruch 1, 2 oder 3, bei dem die Kristallisierung der Alpha-Maltose bei einer Temperatur im Bereich von 60 - 120 °C bewirkt wird, während die Kristallisierung des Beta-Maltosehydrats und die Umsetzung der erhaltenen kristallinen Alpha-Maltose in kristallines Beta-Maltosehydrat bei einer Temperatur im Bereich von 20 - 70 °C bewirkt wird.

Revendications

1. Procédé de préparation de poudre de maltose, comprenant les étapes consistant à concentrer une solution aqueuse de maltose très pur, ayant une teneur en maltose d'au moins 85 % en poids, sur la base de la substance sèche, en un sirop de concentration élevée, ayant une teneur en humidité de moins de 10 % en poids ;
d'abord laisser cristalliser le sirop résultant, de concentration élevée, en alpha-maltose, en présence d'un germe cristallin ; et
laisser cristalliser le mélange résultant en hydrate de bêta-maltose, tout en transformant l'alpha-maltose cristallin résultant en hydrate de bêta-maltose cristallin.
2. Procédé selon la revendication 1, dans lequel ledit sirop de concentration élevée a une teneur en humidité comprise entre 5,0 et 8,0 % en poids.
3. Procédé selon la revendication 1 ou 2, dans lequel l'alpha-maltose cristallin est utilisé seul ou combiné avec de l'hydrate de bêta-maltose cristallin en tant que germe cristallin.
4. Procédé selon la revendication 1, 2 ou 3, dans lequel on effectue la cristallisation de l'alpha-maltose à une température comprise entre 60 et 120 °C, alors qu'on effectue la cristallisation de l'hydrate de bêta-maltose et la transformation de l'alpha-maltose cristallin résultant en hydrate de bêta-maltose cristallin à une température comprise entre 20 et 70 °C.